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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(21) International Application Number: PCT/US92/00613 (22) International Filing Date: 24 January 1992 (24.01.92) (30) Priority data: 651,468 6 February 1991 (06.02.91) US (71) Applicant: MINNESOTA MINING AND MANUFACTURING COMPANY [US/US]; 3M Center, Post Office Box 33427, Saint Paul, MN 55133-3427 (US). (72) Inventors: STEELMAN, Ronald, S. ; Post Office Box 33427, Saint Paul, MN 55133-3427 (US). CRANDALL, Michael, D. ; Post Office Box 33427, Saint Paul, MN 55133-34327 (US). DELGADO, Joaquin ; Post Office Box 33427, Saint Paul, MN 55133-3427 (US).		(74) Agents: ODAR, Thomas, J. et al.; Intellectual Property Counsel, Minnesota Mining and Manufacturing Company, Post Office Box 33427, Saint Paul, MN 55133-3427 (US). (81) Designated States: AT (European patent), AU, BE (European patent), BR, CA, CH (European patent), CS, DE (European patent), DK (European patent), ES (European patent), FR (European patent), GB (European patent), GR (European patent), HU, IT (European patent), JP, KR, LU (European patent), MC (European patent), NL (European patent), SE (European patent). Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
(54) Title: POSITIONABLE ADHESIVE SYSTEM WITH HIGH SHEAR STRENGTH (57) Abstract A positionable aqueous adhesive composition is provided, comprising a blend or mixture of (a) an aqueous suspension of hollow, polymeric, inherently tacky elastomeric microspheres, and (b) an aqueous film-forming pressure sensitive adhesive latex. The adhesive is positionable, in that it allows for positional adjustment after application to a receptor, yet displays high peel adhesion and shear strength.		

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POSITIONABLE ADHESIVE SYSTEM WITH HIGH SHEAR STRENGTHField of the Invention

This invention relates to a positionable
5 adhesive system displaying final elevated shear
strength and peel adhesion, comprising a mixture of
hollow, polymeric, acrylate, infusible, inherently
tacky elastomeric microspheres together with a
polymeric film-forming pressure sensitive latex
10 adhesive.

Background of the InventionDescription of the Related Art

15 Positionable adhesives are those which allow
for placement of an article containing such an adhesive
onto a receptor in an exact position, because the
article can be adjusted relative to the receptor after
initial placement.

20 In some instances, the adhesive can be
designated repositionable or repeatedly reuseable.
Such adhesives exhibit aggressive tack but low peel
adhesion properties, thus allowing repeated
reuseability. Commercial products such as the 3M Brand
25 Post-itTM Notes display such adhesive characteristics.

Positionable adhesive systems, however, as used
herein, generally display high peel adhesion and
elevated shear strength after positioned placement.

Such systems have been disclosed for utility
30 such as in the creation of sign faces or other graphic
systems utilizing a vinyl film. For example, U.S.
Patent Nos. 3,314,838; 3,331,729 and 3,413,168 disclose
the concept of positionability based on a conventional
film-forming adhesive having contained therein
35 frangible glass bubbles. Positionability is obtained
by the fact that the glass bubbles, during initial
application of the coated article,

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allow for incomplete surface contact of the adhesive to the ultimate receptor. Once the article is properly positioned, pressure on the face of the article causes a rupture of the glass bubbles, thus allowing the full surface area to be contacted with the receptor, forming a strong bond. Obviously, once the frangible bubbles are ruptured, any further positionability characteristics are lost. In addition, articles coated with the adhesive containing the frangible bubbles inherently have added manufacturing steps and furthermore require special liners for application.

Reported adhesive systems for repositionable products range from the microsphere adhesives disclosed in U.S. Patent Nos. 3,691,140 and 4,166,152 to pattern-coated conventional adhesive systems. A further definition of an adhesive system containing such microspheres is disclosed in U.S. Patent No. 3,857,731, wherein a binder material is mixed with the microspheres. In some examples in this latter patent, the binder is an acrylate pressure sensitive adhesive. However, in all instances, the adhesion characteristics of the microspheres themselves are not taught to be overshadowed by the pressure sensitive adhesive. In this instance, the binder creates a physical "socket" to mechanically hold the individual microspheres onto a substrate, thus asserting to prevent undesired transfer of the microspheres to a receptor, increasing the retention of desired adhesive features.

U.S. Patent No. 4,735,837 discloses a similar system, wherein solid microspheres are mixed with a binder resin, which is taught to be a pressure sensitive adhesive. In this disclosure, the characteristics of adhesion and repositionability come from the pressure sensitive adhesive rather than the microspheres themselves. In fact, the patent discloses that the microspheres need not have adhesion capabilities at all.

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In EPO 209,337, it is disclosed that the basic deficiency of solid microsphere adhesives is that they exhibit excessive transfer. In this instance, a reactive monomer is included with the adhesive microspheres during their preparation. The monomer remains unreacted during the adhesive polymerization, but subsequently undergoes a reaction with the binder and/or the backing to provide shear values of up to 50 Newtons, claimed to be higher than other microsphere-based systems.

In contrast with the foregoing, we have now discovered that an aqueous pressure sensitive adhesive system can be made utilizing a combination of hollow microspheres which are polymeric, acrylate, infusible and inherently tacky, together with a polymeric film-forming pressure sensitive adhesive. This aqueous blend, when applied to a backing, displays the desired positionability, ease of application, and typically associated with the positionable adhesives discussed above, and in addition displays the high shear strength, high peel adhesion and other performance characteristics normally associated with conventional film-forming pressure sensitive adhesives. In addition, we have found that this system provides for clean removal from most receptors.

Summary of the Invention

In accordance with the invention there is provided a positionable aqueous adhesive composition and article containing same thereon, the composition comprising a blend or mixture of (a) an aqueous suspension of hollow, polymeric, infusible, inherently tacky elastomeric microspheres, and (b) an aqueous film-forming pressure sensitive adhesive latex comprising at least one long chain alkyl acrylate. Preferably, the weight ratio of microspheres to latex,

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based on solids, is from about 12 to 1 to about 39 to 1.

The adhesive is positionable, i.e., can be adjusted positionally after application to a receptor, yet displays high peel adhesion and shear strength, and displays clean removal from most receptors.

Graphic or decorative films, e.g., polyvinyl chloride can be applied to a receptor and positioned accurately, yet final peel adhesion and shear forces will be sufficiently elevated to allow the film to remain in place without movement or deformation.

Detailed Description of the Invention

The hollow polymeric microspheres having utility herein are disclosed in detail in commonly assigned copending application Serial No. 276,767, incorporated herein by reference. Such microspheres comprise at least about 85 parts by weight of at least one alkyl acrylate or alkyl methacrylate ester together with up to about 15 parts by weight of at least one polar monomer, a majority of such microspheres having one or more interior voids. Preferably, the microspheres contain a central cavity at least 10%, and most preferably, 30% of the diameter of the microsphere itself. An aqueous suspension of such hollow microspheres can be prepared by emulsification processes as disclosed in the subject application. Basically, they can be prepared by a two-step process comprised of forming a water-in-oil emulsion of an aqueous solution of one or more polar monomers in oil phase monomers; forming a water-in-oil-in-water emulsion by dispersing the first emulsion into an aqueous phase; followed by initiating polymerization, preferably by the application of heat or radiation. In addition, aqueous suspensions of such microspheres which contain moderately ionized polar monomers may be prepared by a simpler one-step process comprising the aqueous suspension polymerization of at least one alkyl

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acrylate or methacrylate ester monomer and at least one non-ionic polar monomer in the presence of at least one emulsifier capable of producing a water-in-oil emulsion inside the droplets, which is substantially stable during polymerization. Both methods produce an aqueous suspension of monomer droplets which upon polymerization become microspheres, the majority of which contain at least one interior cavity as discussed above.

10 The aqueous pressure sensitive adhesive latex used to blend with the aqueous suspension of hollow microspheres is composed of at least one long chain alkyl acrylate, i.e., containing from 4 to 12 carbon atoms, and preferably a polar monomer.

15 The film-forming pressure sensitive adhesive should be used at a level lower than the interspatial volume of the hollow microspheres. Preferably, the adhesive mixture contains a microsphere to film-forming adhesive ratio of from 39:1 to 12:1. Levels of
20 film-forming adhesive significantly higher than this may produce adhesive coated articles that retain the characteristics of the film-forming pressure sensitive adhesive, detrimental to positionability.

 The aqueous composition can be conventionally
25 applied onto materials such as paper, and vinyl and polyester films, as by knife or notched-bar coating techniques, for example.

 Additives can be included in the mixture to enhance specific objectives, such as the coating of the
30 aqueous system or modifying performance characteristics thereof.

 Anti-foams can be added, especially when the aqueous system of the invention is to be applied as a coating at high rates of speed. One example of such a
35 material is "Foamaster" JMY, a conventional anti-foam agent from Henkel Process Chemicals, Inc.

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Colorants can be added to enhance visual appearance, quality control, etc. Several vendors provide concentrates which add such desirable features.

Chemical crosslinkers, such as polyaziridines, can increase ply adhesion, high temperature shear and cohesive strengths. Such improvements, however, typically can provide a loss in tack and ultimate adhesion.

Viscosity control materials can be added to assist in coating quality and control. Examples include water soluble materials such as various cellulose products, polyacrylic acids, and vinyl alcohols.

The invention will now be described by the use of the following non-limiting examples, wherein all parts are by weight unless otherwise specified.

Example 1

Hollow microspheres are manufactured in accordance with the following procedure:

In a one-liter resin reactor equipped with mechanical stirrer, thermometer and inlet-outlet lines for vacuum and nitrogen, 450 grams of deionized water, 141 grams of iso-octyl acrylate, 9.0 grams of acrylic acid and 0.5 gram of benzoyl peroxide were charged. Vacuum was applied to evacuate the reactor atmosphere and the reactor was purged with argon. The agitation was set to 400 RPM and when the initiator had dissolved, 1.5 grams of ammonium lauryl sulfate (Standapol A, from Henkel AG) were added. The temperature of the reactor was raised to 60°C and maintained for 22 hours, an argon purge being maintained during the polymerization. The suspension was then allowed to cool to room temperature. The reactor was then emptied and the suspension filtered. Optical microscopy showed hollow microspheres from about 4 to about 90 microns in diameter, the majority

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of the microspheres containing a central cavity of at least 30% of the diameter of the microspheres.

This microsphere suspension exhibits a pH typically between 2.0 and 4.0 and has a solids content of approximately 25%. With this procedure, the mean sphere diameter is typically 40-60 microns. (Different vessel proportions or mixing rates will alter the particle size, void diameter, etc.)

An aqueous pressure sensitive adhesive was prepared as follows:

A split-resin flask of 2000 ml. capacity was fitted with a variable speed agitator, condenser, purging tube for introducing nitrogen, and a recording controller. The following materials were added to the flask while purging same with nitrogen:

	404 grams	Distilled water
	1.50 grams	Sodium dodecylbenzene sulfonate
	435 grams	Isooctyl acrylate
20	60 grams	N-tert-octylacrylamide
	0.60 gram	Sodium bicarbonate
	5.0 grams	Sodium styrene sulfonate

The solid N-tert-octylacrylamide was dissolved in the isooctyl acrylate before adding to the flask. The nitrogen purge was continued until the end of the run. The flask and its contents were heated to 50°C, at which temperature an initiator charge of 0.05 gram potassium persulfate and 0.0125 gram sodium metabisulfite was added. The reaction temperature was held at 50°C for approximately 24 hours to complete the polymerization. The resulting latex had no coagulum and the solids content was 54%.

The adhesives as prepared above were combined by adding, to a suitable container, 95.88 parts of the aqueous microsphere suspension at 25% solids; 3.6 parts of the aqueous pressure sensitive adhesive containing 54% solids; 0.52 part of ASE-60, commercially available

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from Rohm & Haas, as a rheological control agent. The pH was then raised with ammonium hydroxide to between 7.5 and 8.5.

The composition was then knife coated onto a polyvinyl chloride film and dried at 200°F for 1 minute to provide a dry coating weight of 19 grams per square meter.

Example 2

The adhesive of Example 1 was duplicated with the exception of utilizing 98.77 parts of the microsphere suspension and 1.23 parts of the pressure sensitive adhesive latex. This was applied to a polyvinyl chloride film as per Example 1.

Example 3

An adhesive blend was prepared by mixing, in a suitable container, 96.23 parts of the aqueous microsphere suspension of Example 1; 2.99 parts of Gelva 2397, an aqueous pressure sensitive adhesive available from Monsanto, containing 65% solids; 0.52 part of ASE-60; followed by raising the pH with ammonium hydroxide to between 7.5 and 8.5; and the addition of 0.26 part of C-E2 color concentrate from Ciba-Geigy to provide opacity.

The composition was coated onto a polyvinyl chloride film at a dry coating weight of 19 grams per square meter, as per Example 1.

Example 4

A film-forming pressure sensitive adhesive was manufactured as follows:

A split-resin flask of 2000 ml. capacity was fitted with a variable speed agitator, condenser, purging tube for introducing nitrogen, and a recording controller. The following materials were added to the flask while purging the flask with nitrogen:

600 grams

Distilled water

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	4.80 grams	Sodium dodecylbenzene sulfonate
	4.80 grams	Nonyl phenol - 10.5 mole ethylene oxide adduct
	16.0 grams	Acrylic acid
5	160.0 grams	Ethyl acrylate
	160.0 grams	Isooctyl acrylate
	64.0 grams	Butyl acrylate

The nitrogen purge was continued until the end of the
10 run. The flask and its contents were agitated at 300
rpm, and was heated to 32°C. At this temperature an
initiator charge of 0.30 gram potassium persulfate and
0.08 gram sodium metabisulfite was added. An
exothermic reaction occurred increasing the temperature
15 to approximately 75°C after which the reaction mixture
was allowed to cool. The resulting latex had no
coagulum and the solids content was 40%.

An adhesive blend was then made by mixing 97.68
parts of the aqueous microsphere suspension of Example
20 1 with 1.57 parts of the film-forming pressure
sensitive adhesive, together with 0.5 part of ASE-60,
adjustment of pH to 7.5 to 8.5 with ammonium hydroxide,
and the addition of 0.25 part of C-E2 color
concentrate.

25 The blend was coated as per Example 1 onto a
polyvinyl chloride film at a coating weight of 19 grams
per square meter.

Example 5

30 A film-forming pressure sensitive adhesive was
made in accordance with Example 4 with the exception
that the monomer mixture was comprised of 320 grams of
ethyl acrylate, 64 grams of butyl acrylate, and 16
grams of acrylic acid. A blend was then prepared by
35 adding 94.46 parts of the aqueous microsphere
suspension of Example 1 to 4.77 parts of the
film-forming pressure sensitive adhesive, again
containing 40% solids, 0.52 part of ASE-60, pH

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adjustment to 7.5 to 8.5 with ammonium hydroxide, and the addition of 0.26 part of C-E2 color concentrate.

Again, the blend was applied onto polyvinyl chloride film at a coating weight of 19 grams per 5 square centimeter as per Example 1.

The foregoing samples were tested for adhesion, both initially and after aging, and dynamic shear was also determined. These results are reported in TABLE 1.

10 For the adhesion tests, a 25.4 mm strip of sample was applied to a test panel using three passes with a 2.27 kg roller. The adhesion was tested by peeling the adhered sample at 180 degrees at a rate of 2.57 mm per minute. The value reported is an average 15 reading over a five second period.

Samples were also aged at room temperature (27°C) for the designated time.

For the dynamic shear test, a .076 mm polyester tape was laminated to a 26.5 mm wide sample to be 20 tested. The samples were adhered to a test panel, typically a painted metal panel, and trimmed such that a 25.7 mm by 25.7 mm contact area was retained. The sample was pulled with an Instron testing machine at a rate of 5 mm/minute. The maximum force required to 25 remove the sample from the test panel is recorded.

TABLE 1

30	Initial Adhesion, Newtons Per Decimeter Width	Adhesion After 24 Hours, Newtons per Decimeter Width	Dynamic shear, Newtons Per Decimeter Square
35 Example 1	20.84	27.50	1176.92
Example 2	22.07	20.97	1117.99

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Example 3	23.67	26.45	1228.06
Example 4	20.67	26.09	993.18
5 Example 5	27.34	27.32	1037.52

Visual observation of the samples showed no sign of adhesive transfer to the receptor test
10 specimens.

For best performance, the blend should be applied at the minimum coating thickness required to obtain a monolayer of microspheres. High coating thicknesses are basically wasteful, while reduced
15 coating thicknesses can result in incomplete surface coverage, and thus reduced peel adhesion and shear values. The preferred coating thickness is represented by the corresponding diameter of the average
microsphere. For example, microspheres made by the
20 aforementioned procedure typically average 40 to 60 microns in diameter, and thus the preferred coating thickness would be in that same range.

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In the Claims

1. An aqueous adhesive composition capable of displaying positionablity, when applied as a layer to a substrate, while maintaining elevated shear strength and peel adhesion, comprising an aqueous blend of

- (a) an aqueous suspension of hollow, polymeric, infusible, inherently tacky elastomeric microspheres; and
- 10 (b) an aqueous film-forming pressure sensitive adhesive latex comprising at least one long chain alkyl acrylate.

2. The composition of claim 1 wherein said 15 microspheres are comprised of at least about 85 parts by weight of at least one alkyl acrylate or methacrylate ester and up to about 15 parts by weight of at least one polar monomer.

20 3. The composition of claim 1 wherein the weight ratio of said microspheres to said latex, on a solids basis, is from about 12 to 1 to about 39 to 1.

4. The composition of claim 1 wherein said 25 latex further comprises at least one polar monomer.

5. An article comprising a substrate, having applied to at least a portion of at least one surface thereof, a coating of a positionable adhesive composition comprising

- (a) an aqueous suspension of hollow, polymeric, infusible, inherently tacky elastomeric microspheres; and
- 35 (b) an aqueous film-forming pressure sensitive adhesive latex comprising at least one long chain alkyl acrylate.

6. The article of claim 5 wherein said substrate is a polyvinyl chloride film.

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7. The article of claim 5 wherein said microspheres are comprised of at least about 85 parts by weight of at least one alkyl acrylate or methacrylate ester and up to about 15 parts by weight of at least one polar monomer.

8. The article of claim 5 wherein the weight ratio of said microspheres to said latex, on a solids basis, is from about 12 to 1 to about 39 to 1.

10

9. The article of claim 5 wherein said latex further comprises at least one polar monomer.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 92/00613

I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all) ⁶		
According to International Patent Classification (IPC) or to both National Classification and IPC		
Int.Cl. 5 C09J7/02; C09J133/08		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System	Classification Symbols	
Int.Cl. 5	C09J	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT⁹		
Category [*]	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
Y	US,A,4 968 562 (J.DELGADO) 6 November 1990 see column 5, line 44 - line 49 see column 8, line 1 - line 7; claims 1-11 ---	1-5
Y,P	EP,A,0 419 020 (3M) see claims 1-4,6,9,10 ---	1-5
<p>[*] Special categories of cited documents: ¹⁰</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"A" document member of the same patent family</p>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
15 JUNE 1992	10.07.92	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	BUSCAGLIONE Y. <i>[Signature]</i>	

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO. US 9200613 SA 57768

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A-4968562	06-11-90	AU-A- 6928391	29-08-91
		EP-A- 0444354	04-09-91
		US-A- 4988567	29-01-91
EP-A-0419020	27-03-91	US-A- 4994322	19-02-91
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		CA-A- 2021958	19-03-91
		JP-A- 3111473	13-05-91

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DK	Denmark			US	United States of America

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AMENDED CLAIMS

[received by the International Bureau on 2 September 1992 (02.09.92);
original claims 1 and 5 amended; remaining claims unchanged (2 pages)]

1. An aqueous adhesive composition capable of displaying positionability, when applied as a layer to a substrate, while maintaining elevated shear strength and peel adhesion, comprising an aqueous blend of:

- (a) an aqueous suspension of hollow, polymeric, infusible, solvent insoluble, acrylate, inherently tacky elastomeric microspheres; and
- 10 (b) an aqueous film-forming pressure sensitive adhesive latex comprising at least one long chain alkyl acrylate having from about 4 to about 12 carbon atoms.

15 2. The composition of claim 1 wherein said microspheres are comprised of at least about 85 parts by weight of at least one alkyl acrylate or methacrylate ester and up to about 15 parts by weight of at least one polar monomer.

20 3. The composition of claim 1 wherein the weight ratio of said microspheres to said latex, on a solids basis, is from about 12 to 1 to about 39 to 1.

25 4. The composition of claim 1 wherein said latex further comprises at least one polar monomer.

30 5. An article comprising a substrate, having applied to at least a portion of at least one surface thereof, a coating of a positionable adhesive composition comprising:

- (a) an aqueous suspension of hollow, polymeric, infusible, solvent insoluble, acrylate, inherently tacky elastomeric microspheres; and
- 35 (b) an aqueous film-forming pressure sensitive adhesive latex comprising at least one long

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chain alkyl acrylate having from about 4 to about 12 carbon atoms.

6. The article of claim 5 wherein said
5 substrate is a polyvinyl chloride film.

7. The article of claim 5 wherein said
microspheres are comprised of at least about 85 parts
by weight of at least one alkyl acrylate or
10 methacrylate ester and up to about 15 parts by weight
of at least one polar monomer.

8. The article of claim 5 wherein the weight
ratio of said microspheres to said latex, on a solids
15 basis, is from about 12 to 1 to about 39 to 1.

9. The article of claim 5 wherein said latex
further comprises at least one polar monomer.